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(54) Process to remove nitrogen from natural gas

(57) Nitrogen is removed from a natural gas feed stream by a cryogenic distillation process in which the feed stream (101) is separated in a distillation column (11); a methane-rich bottoms liquid (103) is recovered as a methane-rich product (104), preferably after being pumped (21) to increase its pressure; a nitrogen-rich overhead vapour (105) is warmed in heat exchange (4) with intermediate vapour stream(s) (112, 114) to at least partially condense said stream(s) for return (113, 115) to the distillation column (11) to provide reflux; and a portion (108) of the warmed nitrogen-rich overhead vapour (105) is utilized as a recycle nitrogen-rich heat pump stream (109, 110, 111) above the critical pressure of nitrogen to provide at least part of the reboil (2) to the distillation column and to produce a mixed vapour-liquid stream, which is returned to the distillation column (11) to provide reflux.

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Description

The present invention relates to a cryogenic process for the removal of nitrogen from feed gas comprising nitrogen and hydrocarbons.

The increasing use of natural gas as a fuel has resulted in a need to remove nitrogen from some natural gas sources, in order to meet Wobbe Index and calorific value specifications, particularly where the gas is delivered into a country's gas transmission system. The nitrogen may either be naturally occurring or resulting from nitrogen injection into oil fields for enhanced recovery.

A particular problem is to design a process for efficient removal of nitrogen from natural gas feed at high pressure (75 to 130 bar absolute; 7.5 to 13 MPa), with relatively small concentrations of nitrogen (5 to 15 mol%), and to produce sales gas at a pressure similar to the feed gas pressure.

A further problem is that, as gas reservoir pressure decays to below the required sales gas pressure (e.g., about 75 bar absolute; 7.5 MPa in the case of the United Kingdom's National Transmission System), feed gas compression needs to be added. This is a relatively expensive investment because it is not utilized fully throughout the life of the nitrogen removal unit (NRU).

Therefore, an object of the present invention is to provide an improved process to remove nitrogen from natural gas feed with low nitrogen content (about 5 to 15 mol%) and at high pressure (75 to 130 bar absolute; 7.5 to 13 MPa). It is a further object of this invention to provide a process for removal of nitrogen from natural gas feed, which is sufficiently flexible to operate at lower feed pressure (25 to 75 bar absolute; 2.5 to 7.5 MPa) while still producing sales gas at higher pressure (about 75 bar absolute; 7.5 MPa), without the need for feed gas compression.

Nitrogen removal from natural gas is usually most economically effected by cryogenic distillation. Numerous cycles have been developed, many based on the concept of double distillation columns as used in air separation. One problem associated with double column cycles is that, at feed nitrogen concentrations less than 25 mol%, the quantity of reflux liquid that can be generated is insufficient to achieve an economic recovery of methane. Another problem is that relatively low concentrations of carbon dioxide and hydrocarbons, such as benzene, hexane and heavier components, would freeze at the cryogenic temperatures associated with the lower pressure column.

GB-B-2208699 describes an improved process that is less energy intensive at low levels of feed nitrogen concentration, in which the separation is effected in two columns with integrated condensation of overhead first column vapour and second column reboil. While this process overcomes the problems mentioned above, it is relatively complicated and expensive.

US-A-4415345 (also EP-A-0090469) describes a process employing a nitrogen heat pump cycle to generate liquid reflux, which is compatible with both single column and double column arrangements. However, this cycle produces methane product at a lower pressure than the natural gas feed and will generally require product gas compression in addition to the nitrogen heat pump compressor. It also limits the column to a low operating pressure of from 15 to 125 psia; (100 to 865 kPa), with heat pump nitrogen at a pressure of 50 to 470 psia (345 to 3,250 kPa), i.e. below the critical pressure. The column pressure is, therefore, dictated by the nitrogen pressure to allow sufficient temperature difference to condense the nitrogen against boiling methane in the column reboiler. This low column operating pressure increases power consumption for product compression.

US-A-4501600 (also EP-A-0132984) discloses a cryogenic rectification process for the separation of nitrogen from natural gas in which a natural gas feed is introduced into a rectification column operating at a pressure of 200 to 450 psia (1.4 to 3.1 MPa) where it is separated into a nitrogen-rich overhead vapour and a methane-rich bottoms liquid. The overhead vapour is partially condensed by indirect heat exchange with vaporizing heat pump fluid and the condensed portion of the overhead vapour is returned to the column as reflux. The heat pump fluid is specified to comprise 0.5 to 60 mole percent nitrogen and 99.5 to 40 mole percent methane and flows in an external closed loop heat pump system. In the exemplified process, an intermediate vapour stream also is condensed against the heat pump fluid and returned to the rectification column as additional reflux.

US-A-4662919 (also EP-A-0233609) discloses a process for separating nitrogen from a pressurized feed containing natural gas and nitrogen in a single distillation column with two side intermediate condensers to form pressurized product streams of nitrogen and natural gas. In the process, the pressurized feed is cooled and separated into separate multiple feeds to the column where the cooled feeds are distilled to form a pressurized nitrogen-rich overhead vapour and a pressurized hydrocarbon-rich bottoms liquid. The overhead vapour is condensed by heat exchange with a first closed loop refrigerant to provide upper reflux to the column; an upper intermediate vapour from the column is condensed in an upper side condenser by heat exchange with the unexpanded overhead vapour to provide an upper intermediate reflux to the column; and a lower intermediate vapour from the column is condensed in a lower side condenser by heat exchange with a second closed loop refrigerant and by heat exchange with the unexpanded overhead vapour to provide a lower intermediate reflux to the column. The upper intermediate vapour is withdrawn from the column between the overhead vapour condenser and the lower side condenser and the lower intermediate vapour is withdrawn from the column between the upper side condenser and the highest feed point to the column. In the

exemplified processes, the first and second closed loop refrigerants are respective portions of an external closed loop methane heat pump system and the distillation column operates at a pressure of 300 to 400 psia (2 to 2.8 MPa). It is stated that since the fractionation above 400 psia (2.8 MPa) approaches the critical pressure of nitrogen, higher pressures are impractical.

US-A-4504295 (also EP-A-0131128) discloses a process for the recovery of methane and nitrogen from a natural gas stream in which a nitrogen rejection stage including a heat pump driven distillation column is integrated with a natural gas liquid stage. In the process, the feed stream is separated into an ethane-rich fraction and nitrogen-rich fraction. After reduction in pressure, the nitrogen-rich fraction, is introduced into a distillation column driven with a closed loop heat pump refrigerant which condenses an overhead reflux stream, condenses an intermediate reflux stream and vaporizes a reboil stream to the distillation column. The ethane-rich fraction is distilled in a second column to provide a methane-rich overhead. In the exemplified process, the distillation column operates at 300 to 400 psia (2 to 2.8 MPa) and the heat pump refrigerant is methane.

The present invention provides a cryogenic process for the removal of nitrogen from a natural gas feed stream comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:

(i) feeding the feed stream to a distillation column providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapour, and at least one intermediate vapour stream; (ii) recovering the methane-rich bottoms as a methane-rich product, preferably after pumping to increase its pressure;
(iii) warming the nitrogen-rich overhead vapour in heat exchange with the at least one intermediate vapour stream to at least partially condense said at least one intermediate vapour stream;
(iv) returning the at least partially condensed intermediate vapour stream to the distillation column, preferably at an intermediate location, to provide reflux;
(v) utilizing a portion of the warmed nitrogen-rich overhead vapour as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapour-liquid stream; and
(vi) returning the mixed vapour-liquid stream to the distillation column, preferably at the top thereof, to provide reflux.

The invention also provides an apparatus for the cryogenic removal of nitrogen from a natural gas feed stream by the process of the invention, the apparatus comprising:

a distillation column for providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapour, and at least one intermediate vapour stream;
means for feeding the feed stream to the distillation column;
means for recovering the methane-rich bottoms as a methane-rich product;
heat exchange means for warming the nitrogen-rich overhead vapour in heat exchange with the at least one intermediate vapour stream to at least partially condense said at least one intermediate vapour stream;
means for returning the at least partially condensed intermediate vapour stream to the distillation column to provide reflux;
means for utilizing a portion of the warmed nitrogen-rich overhead vapour as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapour-liquid stream; and
means for returning the mixed vapour-liquid stream to the distillation column to provide reflux.

One or more intermediate vapour streams can be partially condensed in step (iii) of the process of the invention but it is preferred that a lower intermediate vapour stream is withdrawn at or above the location of the feed point of the natural gas feed stream and returned as reflux above said withdrawal point, and an upper intermediate vapour stream is withdrawn at or above said lower reflux feed point and returned as reflux above the withdrawal point of the upper intermediate vapour stream.

Suitably, the methane-rich bottoms liquid is pumped and then vaporized to increase its pressure before recovery as the methane-rich product.

Usually, the distillation column is reboiled by heat exchange with both the natural gas feed stream and the recycle nitrogen-rich heat pump stream.

In a presently preferred embodiment, the present invention relates to a cryogenic process for the removal of nitrogen from a natural gas feed stream comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:

(a) cooling and at least partially condensing the natural gas feed stream;
(b) further cooling and reducing the pressure of at least part of the natural gas feed stream and feeding it to an

intermediate location of a single distillation column;
(c) removing a methane-rich bottoms liquid from the distillation column, pumping the removed, methane-rich bottoms liquid to increase its pressure, vaporizing the pumped, removed, methane-rich bottoms liquid, and recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product;
(d) removing a nitrogen-rich overhead stream from the distillation column, warming the removed, nitrogen-rich overhead stream to recover refrigeration, and dividing the warmed, removed, nitrogen-rich overhead stream into first and second substreams;
(e) expanding and warming said first substream to recover refrigeration;
(f) warming said second substream, compressing the warmed, second substream, cooling the compressed, second substream, and expanding the cooled, compressed, second substream thereby producing a mixed vapour-liquid stream;
(g) feeding the mixed vapour-liquid stream to the top of the distillation column; and
(h) using at least a part of the refrigeration recovered from warming the nitrogen-rich overhead stream of step (d) to condense at least one intermediate vapour stream from the distillation column to provide intermediate reflux to the distillation column.

Preferably, the natural gas feed stream is divided into first and second portions; said first portion is reduced in pressure and fed to an intermediate location of the distillation column; and said second portion is reduced in pressure, partially vaporized and then fed to the distillation column at a location below the feed for said first portion.

A portion of the warmed nitrogen-rich overhead vapour suitably is expanded and then further warmed to recover refrigeration.

The methane-rich bottoms liquid usually will be subcooled prior to pumping and suitably this subcooling is by heat exchange with a reduced pressure portion of the feed stream.

The recycle nitrogen-rich heat pump stream conveniently is both compressed and, after cooling, subsequently expanded in a compander.

Preferably, the pressure of at least part of the natural gas feed stream is reduced, prior to feeding to the distillation column, with a dense fluid expander.

The distillation column can have an intermediate reboiler/condenser located below the feed point of the natural gas feed stream.

The following is a description by way of example only and with reference to the accompanying drawing of a presently preferred embodiment of the invention.

Figure 1 is a schematic diagram of the process of the present invention.

Referring to the process flow diagram shown in Figure 1, a natural gas feed in line 101, which has been treated to reduce to acceptable concentrations freezing components, such as water and carbon dioxide, is cooled and at least partially condensed in main heat exchanger 1. The natural gas feed will generally contain 5 to 15 mol% nitrogen with the balance being natural gas and impurities and will be at a pressure of 25 to 130 bar absolute (2.5 to 13 MPa), preferably 60 to 80 bar absolute (6 to 8 MPa). The cooled and at least partially condensed natural gas feed is then further cooled and condensed (if not totally condensed in main heat exchanger 1) in reboiler 2, and split into two portions in lines 201 and 202. The major portion in line 202 is subcooled in subcooler 3 before being introduced via line 102 to distillation column 11 through valve 41. The smaller portion in line 201 is reduced in pressure across valve 42 and partially vaporized in subcooler 5 before also being introduced to distillation column 11.

Distillation column 11 operates at a pressure from 10 to 30 bar absolute (1 to 3 MPa), preferably between 15 and 22 bar absolute (1.5 and 2.2 MPa), and separates the natural gas feeds into a methane-rich bottoms liquid stream 103 and a nitrogen-rich overhead vapour stream 105. The nitrogen-rich overhead stream 105 typically contains 2 mol% methane, and the methane-rich bottoms stream 103 has a typical nitrogen concentration of 0.5 mol%, which is generally lower than the required nitrogen content of natural gas that is delivered, for example, to the United Kingdom's National Transmission System (NTS), where concentrations of 4 to 5 mol% are acceptable in gas with parts per million concentrations of carbon dioxide. By reducing the nitrogen content to this low level, which is perfectly feasible in a cryogenic NRU, the quantity of natural gas feed that must be processed is reduced, the final sales gas product being blended from feed gas bypass and NRU product. The UK's NTS specification allows up to 2 mol% CO_2, and with increasing CO_2 content, nitrogen would need to be removed to a lower concentration in the sales gas by processing more gas in the NRU.

Part of the reboil duty for column 11 is provided by heat exchange with the natural gas feed cooling in reboiler 2. The remainder is provided by heat exchange with a recycle, nitrogen-rich heat pump stream also cooling in reboiler 2. The recycle, nitrogen-rich heat pump stream in line 110 is cooled in main heat exchanger 1 and reboiler 2 at a pressure of 35 to 130 bar absolute (3.5 to 13 MPa), preferably between 60 and 80 bar absolute (6 and 8 MPa). The cooled, recycle, nitrogen-rich heat pump stream in line 111 is then expanded in the expansion wheel of compander 32 and introduced into the top of distillation column 11. The expander outlet stream contains 4 to 5% by mass of liquid, and

this is used to provide reflux for the top section of distillation column 11.

The nitrogen-rich overhead vapour in line 105 from the top of column 11, containing about 2 mol% methane, is warmed in condenser 4 and subcooler 3. Condenser 4 provides the bulk of the reflux liquid for distillation column 11 by condensing vapour side streams withdrawn from the column. The lower side stream in line 112 is withdrawn at or above the feed entry location (line 102) and returned as reflux liquid in line 113 which is several equilibrium stages above the withdrawal point (line 112). The upper side stream in line 114 is withdrawn at or above the lower reflux feed point (line 113) and returned in line 115 several equilibrium stages above this withdrawal point. This reflux philosophy is more efficient than a process that provides all of the column reflux liquid at the top of the column, because the majority of the refrigeration required to condense the reflux is provided at the warmer condensing temperatures of the side streams.

After warming in subcooler 3, a portion of the nitrogen-rich overhead vapour in line 106 is expanded in expander 33, providing additional refrigeration to condenser 4 and subcooler 3. This expanded portion is then warmed in main heat exchanger 1 and vented via line 107 to atmosphere. Environmental constraints will generally limit the methane content in vented nitrogen to 2 mol% maximum. The process is capable of achieving much lower methane content, if required, by increasing the recycle nitrogen flow in line 110. Some of the nitrogen-rich vent stream may be used as utility nitrogen for purposes such as cold box purge and adsorber regeneration. The remaining nitrogen-rich overhead vapour is warmed in main heat exchanger 1, compressed in compressor 31, passed via line 109 to the compressor wheel of compander 32 and cooled in cooler 6 to form the recycle, nitrogen-rich stream to main heat exchanger 1 in line 110.

Methane-rich bottoms liquid from column 11 is subcooled in subcooler 5 before being increased in pressure by pump 21. Subcooler 5 minimizes the elevation which distillation column 11 is required to be above pump 21 in order to provide the necessary net positive suction head (NPSH) at the pump suction, particularly, if there is a large turndown requirement where heat leak into the pump suction piping could cause cavitation at turndown. The pumped liquid is then vaporized in main heat exchanger 1 to be delivered as sales gas product at a pressure of 25 to 130 bar absolute (2.5 to 13 MPa), preferably 60 to 80 bar absolute (6 to 8 MPa).

The process achieves a very high methane recovery, typically about 99.8 mol%, because the methane content in the vent nitrogen can be reduced to less than 2 mol%.

Table 1 summarizes a mass balance for a typical application of this invention.

TABLE 1

Stream		101	102	103	104	105	106	107	108	109	110	111	112	113	114	115
Pressure	bar abs	78.3	74.4	18.8	79.4	18.6	18.1	1.2	17.8	60.2	76.6	74.9	18.7	18.7	18.7	18.6
	[kPa]	7,830	7,440	1,880	7,940	1,860	1,810	120	1,780	6,020	7,660	7,490	1,870	1,870	1,870	1,860
Temperature	deg C	30	-113	-108	21	-158	-109	21	21	30	30	-108	-119	-142	-137	-162
Flowrate	kg-mol/h	100	98.74	91.61	91.61	84.71	8.39	8.39	76.33	76.33	76.33	76.33	13.2	13.2	12.59	12.59
Composition																
Hydrogen	mol%	0.052	0.052			0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.155	0.155	0.239	0.239
Helium	mol%	0.031	0.031			0.37	0.37	0.37	0.37	0.37	0.37	0.37	0.090	0.090	0.138	0.138
Nitrogen	mol%	8.582	8.582	0.49	0.49	97.01	97.01	97.01	97.01	97.01	97.01	97.01	30.413	30.413	69.685	69.685
Carbon dioxide	mol%	0.005	0.005	0.005	0.005											
Methane	mol%	87.487	87.487	95.309	95.309	2.0	2.0	2.0	2.0	2.0	2.0	2.0	69.338	69.338	29.938	29.938
Ethane	mol%	2.847	2.847	3.108	3.108								0.004	0.004		
Propane	mol%	0.618	0.618	0.675	0.675											
Butanes	mol%	0.314	0.314	0.343	0.343											
Pentanes	mol%	0.051	0.051	0.056	0.056											
n-Hexane	mol%	0.011	0.011	0.012	0.012											
n-Heptane	mol%	0.002	0.002	0.002	0.002											

Several modifications of the above-described process are possible within the scope of the invention, including:

The number of side streams that are condensed in condenser 4 could be increased to three or more if the resulting reduction in power consumption warranted the additional complexity. Alternatively, the system could be simplified by condensing only one side stream.

All or part of the vent nitrogen can be produced at a higher pressure and used as a by-product by increasing expander 33 outlet pressure or by the removal of a nitrogen containing stream prior to the inlet to compressor 31, prior to the inlet to the compressor wheel of compander 32, or from downstream of cooler 6. This may result in the elimination of expander 33 from the process. It is possible to eliminate expander 33 in any event by increasing the refrigeration produced by compander 32, although this is less efficient.

Expander 33 could be moved to provide refrigeration at a warmer part of the process, e.g., around exchanger 1. This could be beneficial where the feed pressure was much lower than the required sales gas product pressure.

In addition to the vent nitrogen, all or some of the recycle heat pump nitrogen could be expanded in expander 33. This would reduce the flowrate of the recycle heat pump nitrogen, but would also reduce the inlet pressure to compressor 31 and, thus, possibly require two (2) differing pressure feeds to the compressor.

It is possible to improve the process efficiency by expanding the feed to the column in a dense fluid expander, rather than valve 41. The expansion work could be recovered in a suitable device, such as an electricity generator, and the refrigeration produced would reduce the refrigeration required from compander 32.

Subcooler 5 could be eliminated and the required pump NPSH developed by increasing the elevation difference between the column sump and the pump suction.

Part of the feed could be subcooled to a lower temperature in subcooler 3, rather than subcooling all of the feed, that is expanded across valve 41, to a warmer temperature. This colder feed could then be introduced to column 11 a few stages higher than the remaining feed from upstream of subcooler 3.

The column system could be modified to include an intermediate reboiler between the bottom of the column and the main feed stage. This may be appropriate for higher feed nitrogen concentrations.

The position of compander 32 could be changed so that recycle nitrogen was compressed in the compressor wheel of the compander before being compressed in compressor 31. This would be determined by the optimum machinery configuration.

Liquid methane from the bottom of column 11 could be further processed to recover a natural gas liquids product.

The process could be modified to recover a helium-rich stream from the overhead vapour from column 11, where there was sufficient helium in the natural gas feed to make this economically attractive.

The process could be operated with a much higher methane content in the vent nitrogen for possible use as a fuel stream with a consequent reduction in power consumption.

The exemplified embodiment of the invention provides a process cycle with only slightly higher power consumption than the efficient cycle described in GB-B-2,208,699, but which is much simpler and has a significantly lower capital cost.

The refrigeration provided by the expansion wheel of compander 32 and expander 33 is sufficient to compensate for pump work, heat leak and temperature difference at the warm end of main heat exchanger 1 and enables the sales gas product to be delivered at a similar pressure to the feed with no need for any product compression. The expansion work of the recycle nitrogen is efficiently recovered in the compressor wheel of compander 32.

If the feed pressure reduces over a period of time, for example, due to decay of gas reservoir pressure, the sales gas can still be produced at the required pressure simply by increasing the refrigeration provided by the expansion wheel of compander 32 beyond what is required for the column reflux liquid. This compensates for the reduced Joule-Thomson refrigeration that is available from the lower pressure feed. By this method, sales gas can be produced at, for example, 79 bar absolute (7.9 MPa) with the feed gas pressure as low as 25 bar absolute (2.5 MPa). Operation of the NRU is less efficient at feed gas pressures much below the required sales gas pressure, and capacity will be reduced because all of the feed gas will need to be processed in the NRU because there can be no bypass. Also, the size of compander 32 will limit production. However, this gives the plant operator the choice of whether or not to invest in feed gas compression and certainly postpones the date at which it becomes economically viable to purchase or lease this compression system.

The problem of freezing carbon dioxide and heavy hydrocarbons is mitigated by the single column process, operating at high pressure, because the freezing components are recovered in the bottom section of the column where the temperature is higher. The closest approach to freezing generally occurs at the feed inlet to the column, downstream of valve 41. The process is tolerant to significantly higher concentrations of carbon dioxide and heavy hydrocarbons than a double column process.

The exemplified embodiment of the present invention differs from prior art by, inter alia,:

(i) Separating nitrogen from methane in a single column where refrigeration for column reflux is provided by a nitrogen-rich heat pump system operating above the critical pressure of nitrogen, i.e. at a pressure of 35 to 130 bar absolute (3.5 to 13 MPa) preferably between 60 and 80 bar absolute (6 and 8 MPa), thereby permitting a higher

column operating pressure of 10 to 30 bar absolute (1 to 3 MPa), preferably between 15 and 22 bar absolute (1 and 2.2 MPa). The column is reboiled by indirect heat exchange with both the nitrogen-rich heat pump stream and the feed stream.

(ii) Expanding the nitrogen-rich heat pump stream in a compander to provide an outlet stream with up to 10% liquid, preferably 4 to 5% liquid, thereby directly providing reflux for the top section of the column and efficiently recovering the expansion work.

(iii) Condensing side streams of column vapour to provide the majority of the reflux liquid at warmer temperatures by indirect heat exchange with the overhead vapour from the column, which comprises the nitrogen-rich heat pump stream and the nitrogen-rich vent stream.

(iv) Providing sufficient refrigeration with the compander 32 and, where included, the vent nitrogen expander 33 to produce, by pumping, all of the sales gas product at a pressure similar to, or higher than, the natural gas feed pressure and avoiding the need for a product compressor.

(v) Subcooling the feed against recycle heat pump nitrogen in subcooler 3 prior to introduction to the distillation column.

(vi) Subcooling the column bottoms liquid prior to pumping in order to provide sufficient NPSH without elevating the column relative to the pump suction. This is particularly beneficial where height limitations are imposed for environmental reasons.

It will be appreciated that the invention is not restricted to the specific details of the embodiment described above and that numerous modifications and variations can be made without departing from the scope of the invention as defined in the following claims.

Claims

1. A cryogenic process for the removal of nitrogen from a natural gas feed stream (101) comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:

   (i) feeding the feed stream (101) to a distillation column (11) providing a methane-rich bottoms liquid (103), a nitrogen-rich overhead vapour (105), and at least one intermediate vapour stream (112,114);
   (ii) recovering the methane-rich bottoms as a methane-rich product (104);
   (iii) warming (4) the nitrogen-rich overhead vapour (105) in heat exchange with the at least one intermediate vapour stream (112,114) to at least partially condense said at least one intermediate vapour stream (112,114);
   (iv) returning the at least partially condensed intermediate vapour stream (113,115) to the distillation column (11) to provide reflux;
   (v) utilizing a portion (108) of the warmed nitrogen-rich overhead vapour (105) as a recycle nitrogen-rich heat pump stream (109,110,111) above the critical pressure of nitrogen to provide at least part of the reboil (2) to the distillation column (11) and to produce a mixed vapour-liquid stream; and
   (vi) returning the mixed vapour-liquid stream to the distillation column to provide reflux.

2. A process as claimed in Claim 1, wherein, in steps (iii) and (iv), a lower intermediate vapour stream (112) is withdrawn at or above the location of the feed point of the natural gas feed stream and, after at least partial condensation (4), returned (113) above said withdrawal point to provide reflux, and an upper intermediate vapour stream is withdrawn at or above said lower reflux feed point and, after at least partial condensation (4), returned (115) above the withdrawal point of the upper intermediate vapour stream to provide reflux.

3. A process as claimed in Claim 1 or Claim 2, wherein the distillation column (11) is reboiled by heat exchange (2) with both the natural gas feed stream (101) and the recycle nitrogen-rich heat pump stream (110).

4. A cryogenic process for the removal of nitrogen from a natural gas feed stream as claimed in Claim 1 comprising:

   (a) cooling and at least partially condensing (1) the natural gas feed stream (101);
   (b) further cooling (2 & 3) and reducing the pressure (41) of at least part (202) of the natural gas feed stream

(101) and feeding it to an intermediate location of a single distillation column (11);
(c) removing a methane-rich bottoms liquid (103) from the distillation column (11), pumping (21) the removed, methane-rich bottoms liquid to increase its pressure, vaporizing (1) the pumped, removed, methane-rich bottoms liquid, and recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product (104);
(d) removing a nitrogen-rich overhead stream (105) from the distillation column (11), warming (4) the removed, nitrogen-rich overhead stream (105) to recover refrigeration, and dividing the warmed, removed, nitrogen-rich overhead stream (105) into first and second substreams (106,108);
(e) expanding (33) and warming (4) said first substream (106) to recover refrigeration;
(f) warming (1) said second substream (108), compressing (31,32) the warmed, second substream (108), cooling (2) the compressed, second substream (110), and expanding (32) the cooled, compressed, second substream (111) thereby producing a mixed vapour-liquid stream;
(g) feeding the mixed vapour-liquid stream to the top of the distillation column (11); and
(h) using at least a part of the refrigeration recovered from warming (4) the nitrogen-rich overhead stream (105) of step (d) to condense (4) at least one intermediate vapour stream (112,114) from the distillation column (11) to provide intermediate reflux to the distillation column (11).

5. A process as claimed in any one of the preceding claims, wherein the natural gas feed stream (101) is divided into first and second portions (202,201); said first portion (202) is reduced in pressure (41) and fed to an intermediate location of the distillation column (11); and said second portion (201) is reduced in pressure (42), partially vaporized (5) and then fed to the distillation column (11) at a location below the feed for the said first portion (202).

6. A process as claimed in any one of the preceding claims, wherein a portion (106) of the warmed nitrogen-rich overhead vapour (105) is expanded (33) and then further warmed (4).

7. An apparatus for the cryogenic removal of nitrogen from a natural gas feed stream (101) by a process as claimed in Claim 1, the apparatus comprising:

a distillation column (11) for providing a methane-rich bottoms liquid (103), a nitrogen-rich overhead vapour (105), and at least one intermediate vapour stream (112,114);
means(101,201,202) for feeding the feed stream to the distillation column (11);
means (103,104) for recovering the methane-rich bottoms as a methane-rich product;
heat exchange means (4) for warming the nitrogen-rich overhead vapour (105) in heat exchange with the at least one intermediate vapour stream (112,114) to at least partially condense said at least one intermediate vapour stream (112,114);
means (113,115) for returning the at least partially condensed intermediate vapour stream to the distillation column (11) to provide reflux;
means (2) for utilizing a portion (108) of the warmed nitrogen-rich overhead vapour as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column (11) and to produce a mixed vapour-liquid stream; and means (111) for returning the mixed vapour-liquid stream to the distillation column (11) to provide reflux.

8. An apparatus as claimed in Claim 7, wherein the heat exchange means (4) at least partially condenses a lower intermediate vapour stream (112) withdrawn from the distillation column (11) at or above the location of the feed point of the natural gas feed stream (101) and an upper intermediate vapour stream (114) withdrawn at or above the feed point at which at least partially condensed lower intermediate vapour stream (113) is returned to the distillation column (11) and the means (113,115) for returning at least partially condensed intermediate vapour to the distillation column (11) to provide reflux returns both said at least partially condensed intermediate streams to the distillation column (11) at locations above their respective withdrawal points.

9. An apparatus as claimed in Claim 7 or Claim 8, comprising means (2) for reboiling the distillation column (11) by heat exchange with both the natural gas feed stream (101) and the recycle nitrogen-rich heat pump stream (110).

10. An apparatus as claimed in Claim 7 comprising:

(a) means (1) for cooling and at least partially condensing the natural gas feed stream (101);
(b) means (2) for further cooling and reducing the pressure of at least part (201) of the natural gas feed stream (101) and feeding it to an intermediate location of a single distillation column (11);

(c) means (103) for removing a methane-rich bottoms liquid from the distillation column (11);
(d) means (21,1) for pumping the removed, methane-rich bottoms liquid to increase its pressure, vaporizing the pumped, removed, methane-rich bottoms liquid, and recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product (104);
(e) means (105,4,3,106,108) for removing a nitrogen-rich overhead stream from the distillation column (11), warming the removed, nitrogen-rich overhead stream to recover refrigeration, and dividing the warmed, removed, nitrogen-rich overhead stream into first and second substreams (106,108);
(f) means (33,4) for expanding and warming said first substream (106) to recover refrigeration;
(g) means (1,31,32) for warming said second substream (108), compressing the warmed, second substream, cooling the compressed, second substream, and expanding the cooled, compressed, second substream thereby producing a mixed vapour-liquid stream;
(h) means (111) for feeding the mixed vapour-liquid stream to the top of the distillation column (11); and
(j) means (4) for using at least a part of the refrigeration recovered from warming the nitrogen-rich overhead stream (105) in means (e) to condense at least one intermediate vapour stream (112,114) to provide intermediate reflux to the distillation column (11).

11. An apparatus as claimed in any one of Claims 7 to 10, comprising means (201,202) for dividing the natural gas feed stream into first and second portions; means (41) for reducing the pressure of said first portion; means (102) for feeding said reduced pressure first portion to an intermediate location of the distillation column (11); means (42) for reducing the pressure of said second portion; means (5) for partially vaporizing said reduced pressure second portion; and means (201) for feeding said partially vaporized second portion to the distillation column (11) at a location below the location of the feed for the said first portion.

12. An apparatus as claimed in any one of Claims 7 to 11, comprising means (33) for expanding a portion of the warmed nitrogen-rich overhead vapour (105) and means (4) for further warming said expanded portion.

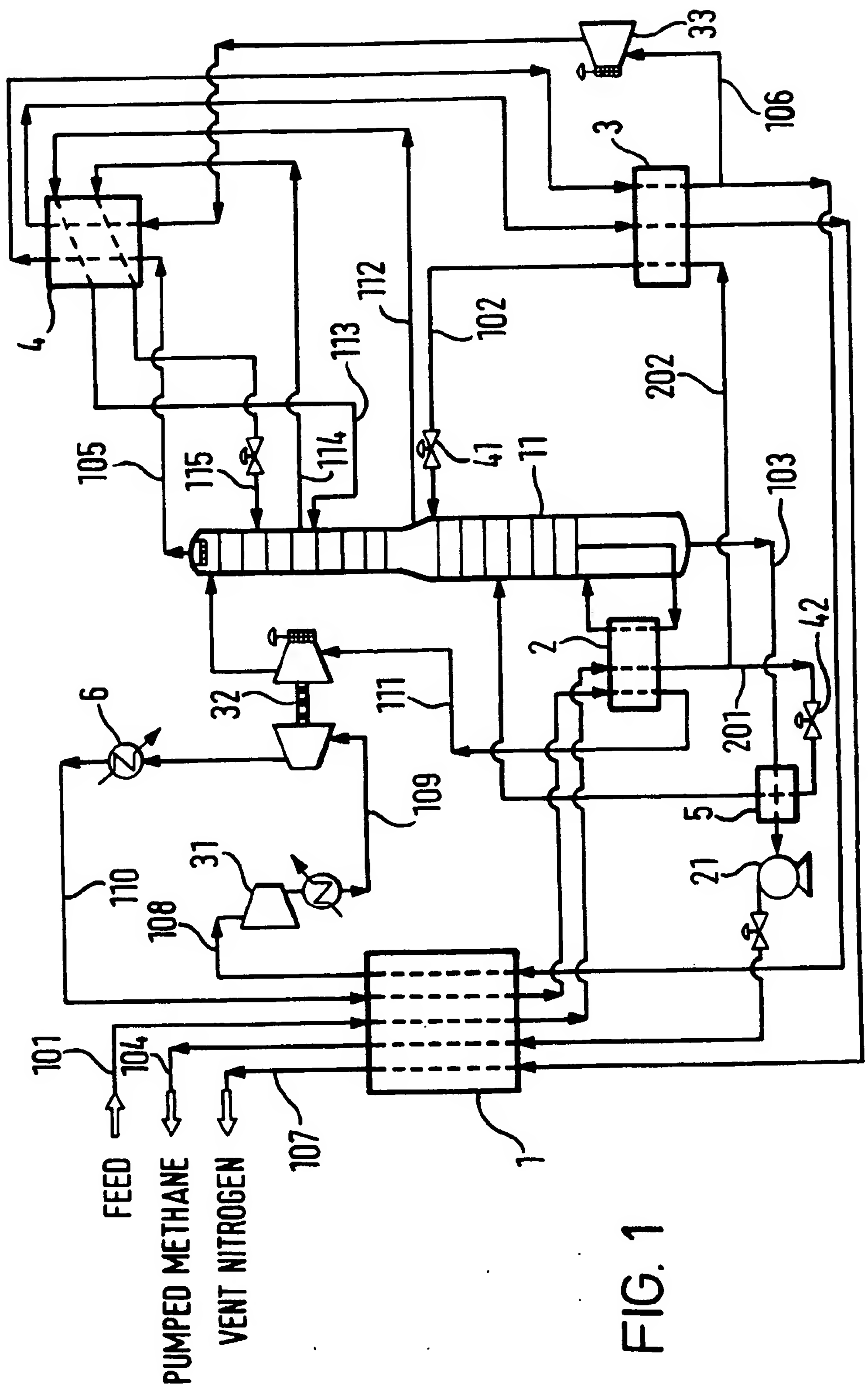
FEED
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FIG. 1

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 96 30 0725

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A,D	US-A-4 662 919 (DAVIS) * claims; figures * ---		F25J3/02 F25J3/08 C10L3/10
A,D	EP-A-0 132 984 (UNION CARBIDE) * claims; figure * ---		
A,D	EP-A-0 090 469 (UNION CARBIDE) * claims; figures * ---		
A	FR-A-2 682 964 (ELF AQUITAINE) * claims; figures 3-5 * -----		
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			F25J C10C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	30 May 1996	Meertens, J

CATEGORY OF CITED DOCUMENTS

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